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In situ observation of crack propagation in ESP (engineered stress profile) glass

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Abstract

Important features of the ESP (engineered stress profile) glasses are the crack arrest and multiple cracking phenomena that occur even in an unstable stress field. In this work a detailed "in situ" observation of crack observation and analysis was performed with the aim to examine crack propagation in detail and relate it to the residual stress field produced by ion exchange and to the final mechanical performances of the material. The results showed that the peculiar residual stress field with a maximum below the surface is responsible for the formation of a multitude of stable cracks on the tensile surface of the glass that evolved into through-thickness flaws. The propagation within the material is limited by the increasing compressive residual stress, which also leads to kinking of the cracks in a direction parallel to the surface. The observed fracture phenomena are also responsible for a shielding effect that makes the measured failure resistance of ESP glass larger than predicted by simplistic single crack models.

Keywords: Glass; Fracture; Crack arrest; Residual stresses; Stable crack growth; *R*-curves

1. Introduction

Recent research activities carried out jointly at The Pennsylvania State University in USA and at the University of Trento in Italy pointed out that two-step ion-exchange treatments can generate a unusual residual stress field on the surface of silicate glasses that allows stable propagation of surface defects before final failure [1–4]. The glasses obtained in this way have been named as ESP glass *i.e.* "*engineered stress profile glass*". These results represent a new approach in the strengthening of glass. In contrast to the traditional ion-exchange approach, where the aim is to increase and deepen the compressive stress on the surface, the key feature in this new approach is to carefully design the residual stress profile in such a way as to move the maximum compression away from the external surface and to carefully control the stress gradient in the surface region. This design of the stress profile leads to some important attributes in the mechanical behavior. As with

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the traditional ion-exchange processes, the compressive stresses still give rise to significant strengthening. The difference is that the stress gradient in ESP glass is such that cracks can be arrested without causing failure even in normally unstable stress fields, *i.e.* those associated with applied stress intensity factors that increase as cracks grow. This effect leads to a reduction in strength variability and to multiple cracking prior to the final failure that 'warns' of the impending failure. Moreover, the strength of ESP glass is less sensitive to surface damage and to the environment. So far, ESP glasses have been produced with both soda lime and soda alumina silicate glasses.

One of the more interesting features of ESP glass is the crack arrest phenomenon in unstable stress fields, such as those associated with bending, mainly because it occurs in completely brittle materials like silicate glasses. In the present work, the results of a detailed fractographic study are presented. The aim is to observe and analyze the crack arrest phenomenon in detail and to relate this to the residual stress profile and the fracture resistance of the material.

2. Experimental procedure

Three different silicate glasses are considered in this work for the production of ESP glass. The chemical composition is shown in Table 1. The first glass (Planilux, Saint Gobain), labelled as S, is a typical soda-lime-silica float glass from a commercial source. The second glass (CE120, Pilkington), labelled as P, is an alkali-silicate glass made by the float process on Pilkington's mini electric melting unit. It has been specially formulated for ultra fast chemical strengthening with improved stress and exchange depth properties compared to soda-lime glasses. The last glass (code 0317, Corning), labelled as C, is a soda-alumina silicate glass, specifically produced to obtain deep penetration depth during the ion-exchange process. The glass transition temperature, T_g, of the glasses measured by differential scanning calorimeter (DSC) method [5] is also reported in Table 1.

As-received glass plates with thickness of about 3 mm were cut into ≈50 mm × 10 mm bars. The edges were chamfered and polished using silicon carbide paper and diamond paste with grain size of 3 μm in order to remove macroscopic defects introduced during the cutting procedure. The samples were then annealed for 8 h at temperature 40 °C below the T_g to remove any residual stress. A cooling rate of 40 °C/h was used in each case.

Batches of 25 specimens were subjected to ion-exchange treatments in a nitrate salt bath using a semi-automatic furnace (LEMA TC 20A, Parma, Italy). The samples were placed into a stainless steel frame, which was inserted into the furnace and maintained above the salt bath for 5 min before immersion into the liquid. At the end of the treatment, the frame was automatically raised and kept above the salt for 25 min to allow draining and initial gradual cooling. Then, the frame was extracted from the furnace and cooled in calm air. Finally, the treated glass samples were carefully cleaned with de-ionized water and dried.

Each glass was subjected to a specific treatment depending on the composition and the transition temperature. The ion-exchange conditions, selected on the basis of previous works [3,4], are reported in Table 2. Some samples were subjected only to the first treatment in KNO_3, aiming to produce a deep compressive residual stress layer on the glass surface. Other samples were subjected also to the second treatment whose intent was to partially reduce the K^+ ion concentration just near the surface of the specimen, thus relieving part of the compressive residual stress, as required for ESP glasses [1–4].

The residual stress profile produced by ion exchange was determined using a technique based on the measurement of the curvature induced by the removal of successive material layers from one of the faces of the ion exchanged specimens [6].

Table 1
Chemical composition (wt%) and glass transition temperature (T_g)

Glass	SiO_2	Na_2O	CaO	K_2O	MgO	Al_2O_3	Other	T_g (°C)
S	71	13	10		4	1	1	557
P	67	14		4	9	5	1	585
C	62	13		4	3	16	2	630